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Formulation & Storage Studies on Hydrazine-Based Gelled Propellants

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ABSTRACT

The current trend in the area of high energy storable liquid rocket propellant research is to develop gelled/metallized systems and to explore the feasibility of their application in rocket motors. The idea stems from the fact that the conversion of a conventional liquid propellant to a gelled state and its subsequent metallization has the potential to significantly enhance the performance and density specific impulse. However, it is mandatory that the gelation of the liquid propellant be brought about with a minimum concentration of gellant and the gel formed should show a shear-thinning behaviour and good storage life. Keeping this in view, an experimental program to prepare thixotropic gels of hydrazine and its methyl substituted derivatives like monomethyl hydrazine and unsymmetrical dimethyl hydrazine was conducted under ambient conditions. The gellants investigated included particulate, chemical and synthetic materials. The gelation of the liquid fuels could be brought about with gellant concentration as low as 1 wt per cent in some cases. Metallized gels using up to 40 wt per cent of Al/Mg metal powders could be formulated. These heterogeneous systems showed a reduction in critical gellant concentration with degree of metallization. Further, less gelation time is observed if the gellant concentration is kept fixed and metal content is increased. The storage studies on gelled systems conducted over a period of three months showed good stability. The metallized systems, however, showed the settling of metal powders in meagre concentration which does not pose a major problem as these gels could be re-homogenized with stirring.

1. INTRODUCTION

Liquid propellant rocket engines deserve the credit for most of the magnificent space achievements. However, there is an ever increasing demands for developing high energy liquid bipropellant systems to meet growing mission requirements. The performance of the liquid and cryogenic fuel systems can be substantially improved by using low molecular weight metal powders like aluminium and magnesium in conjunction with the base fuel. A suitable approach to formulate these metallized fuels is to first gellify the fuel and then disperse the fine metal powders in the bulk. The homogeneous distribution of the metal particles is achieved by proper mixing while the particle suspension requirement can be ensured by simultaneously gelling the metallized fuel. Besides the improved performance[1-4], the gelled propellants also provide additional benefits to a propulsion engineer[5-7] including reduction in boil-off losses and sloshing, better operational safety and fuel storability and rocket engine restart under zero gravity conditions.

Considerable interest has been evinced in the recent past towards the application of these heterogeneous fuel systems in advanced aerospace missions[8-11]. However, the details about their rheological behaviour, ignition and combustion characteristics, feed system design, tank expulsion, safety and handling characteristics have to be precisely understood before their use in future missions. A significant research and development effort[12-18] in the above mentioned areas has been made which reflects immense promise for application of these high energy metallized fuel systems.

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The shear-thinning behaviour and combustion efficiency of heterogeneous fuels primarily depend on their formulation. It is desired that such gelled systems should exhibit thixotropic character and their viscosity be brought down as close to the parent propellant level as possible when it enters the combustion chamber. Most of the gellants reported in literature gellify the base propellant at unacceptably high concentrations which is detrimental from the performance point of view. Very little work is reported in open literature about the effectiveness of various gellants for hydrazine based fuels which may work at low gellant levels. Keeping this in view, the present study aims to explore the development of suitable thixotropic gels of hydrazine, monomethyl hydrazine and unsymmetrical dimethyl hydrazine and suitable gellants and to report their critical concentrations for virgin and metallized gels, effect of gellant grades on gelation and gel storage behaviour. Efforts have also been made to explain the efficacy of typical gellants with a particular fuel.

2. EXPERIMENTAL DETAILS

2.1 Materials

Hydrazine hydrate (HH) (80 per cent) procured from M/s Loba-Chemie, India, monomethyl hydrazine (MMH) obtained from Vikram Sarabhai Space Centre, Thiruvananthapuram and unsymmetrical dimethyl hydrazine (UDMH) collected from Defence Research and Development Laboratory, Hyderabad, were used to formulate gels as such without any further purification.

A particulate gelling agent silica fumed, Cab-O-Sil (BDH, England) was tried. The chemical gelling agents like methyl cellulose (MC) - low (LS1MC & LS2MC) and high substitution (HSMC), hydroxyethyl cellulose (HEC, Schuchardt Munchen, Germany), polyacrylic acid (25 per cent solution, mol. wt. approx. 2,30,000), polyacrylamide (PAA, mol. wt. over 5×10^6), carboxymethyl cellulose (CMC) of BDH England, pectin (Koch-light, England), polyvinyl alcohol (PVA, Amrut, India), hydroxy propyl methyl cellulose (HPMC, Colorcon, England) and other chemicals of good quality were used for preparing synthetic gelling agents of ASPAA type. Aluminium and magnesium metal powders of 20 μm and 60 μm particle size respectively have been used.

2.2 Preparation of Gels

The gels of HH, MMH and UDMH have been prepared by adding known amounts of gellants to a fixed volume of liquid fuel, followed by mixing operation for 2 min duration. The gelation time in each case has been found out by slightly tilting the stoppered tubes (L = 12 cm, I.D. = 1.5 cm) and observing the no-flow condition at regular time intervals.

Hydrazine hydrate could be gelled by gelling agents like pectin, PAA, PVA, CMC, HEC and ASPAA-II whereas MMH was successfully gellified by LS1MC, LS2MC, HSMC, HEC, HPMC and ASPAA-II in requisite concentrations. The gels of UDMH have been prepared using gellants such as Cab-O-Sil, PAA, LS1MC, LS2MC, HSMC and HEC. Subsequently, MMH gelled fuel systems with HPMC and ASPAA gellants were chosen for metallization due to their low gellant concentration requirement and better gel quality.

2.3. Preparation of Metallized MMH Gels

Metallized gels of MMH were prepared by incorporating 10-40 wt per cent of *Al* (20 μm) and *Mg* (60 μm) powders. The gelation in each case was achieved by using critical or near critical concentrations of respective gellants. The metallized gels using cellulose derivatives as gellants were prepared by mixing the gellant with metal powder thoroughly followed by their dispersion in MMH with constant stirring to avoid aggregate formation. However, the synthetic gellants were first mixed with the liquid fuel followed by dispersion of metal powder (10-40 wt per cent) to prevent their settling as the viscosity of parent liquid increases rapidly after gellant addition.

2.4 Ageing Studies of MMH Gels

Gels of MMH using the most effective gellants like HPMC and ASPAA were prepared in QF stoppered specimen tubes as discussed earlier. Virgin and metallized gels were then stored under ambient conditions for a period of 15 weeks with simultaneous recording of observations.

3. RESULTS AND DISCUSSION

3.1 Hydrazine-based Virgin Gelled Systems

The data on gelation studies of HH using gelling agents of different nature have been recorded in Table 1. The critical concentrations (wt per cent) of gellants recorded therein represent the minimum quantity which brings about the gelation. It is noteworthy that MC and Cab-O-Sil known for their gel forming ability, do not gellify HH at all at least up to 48 hr. The other cellulose derivatives such as HEC and CMC successfully gel HH in 1 and 24 hr respectively at the same (5 wt per cent) critical concentration. Pectin which is polygalacturonic acid (linked 1,4) with partially methylated carboxyl groups is seen to be effective in gelling HH in only 10 min while non-carbohydrate gellants such as PVA and PAA appear to be very attractive as only 1.25 wt per cent of them is sufficient to convert HH into a gel state. The most attractive gellant from propulsion viewpoint, however, is found to be ASPAA-II whose only 0.5 wt per cent concentration brings about the gelation of HH and that too within a minute. The other gellants like low and high substituted MC and HPMC1OOK are seen to be ineffective as far as HH gelling is concerned.

Table 1. Gelling ability of gellants in case of HH, MMH and UDMH fuels

S. No.	Gellant	Liquid Fuel		
		HH	MMH	UDMH
1.	Cab-o-Sil	x	x	(5, 45)*
2.	Pectin	(6, 10)	x	x
3.	12.5% of Aq. Solution of PAA	(1.25, 240)	x	(0.25,30)
4.	PVA	(1.25,120)	x	x
5.	LS1MC	x	(9,27)	(4.48,20)
6.	LS2MC	x	(11,30)	(5,8)
7.	HSMC	x	(8,180)	(2,10)
8.	CMC	(5,24 hr)	x	x
9.	HEC	(5,60)	(7,45)	(7,45)
10.	HPMCl00k	x	(2.75,12)	x
11.	ASPAA-II	(0.50,1)	(1.75.1)	x

* First quantity within parenthesis represents wt% of gellant and second one denotes gelation time in minutes at 25±2°C.

x No gelation occurs till 24 hr using 5 wt% gellant.

It is promising to note that gellants like ASPAA-II, PVA and PAA gellify HH in concentration levels ≤ 1.25 wt per cent. It may be mentioned that these gellants are capable of forming aqueous gels easily and thus HH which contains 20 per cent of water, v/v, is effectively gelled by them. The efficiency of CMC and HEC in HH gelation, however, can be explained on the structural grounds of the gellants and fuel entity. The CMC (sodium salt) with free carboxyl and HEC, with hydroxyl group in the side chain will obviously have better chances of interaction with hydrazine to form hydrogen bond and bring about its gelation. The LS1MC, however, remains ineffective in this case possibly because of the presence of methoxyl group directly attached to the ring hindering the hydrogen bonding to some extent. It has also been observed that MC tends to cottonize in the presence of water and hence its solvation and uniform dispersion in the fuel is adversely affected.

The authors have already carried out a number of investigations and reported their results not only on the gelation of UDMH but also related to the exploration of the possibility of use of metallized UDMH gels as a potential heterogeneous liquid fuel. Further attempts have, nevertheless, been made to search for new gellants effective at lower concentrations and imparting better gel quality to the parent liquid. The various promising gellants for UDMH have also been enlisted in Table 1 alongwith their respective critical concentrations and gelation times. It is found that gelling agents such as pectin, PVA, CMC and ASPAA-II which have successfully gelled HH are ineffective in the case of UDMH. It may, therefore, be concluded that except the Cab-O-Sil, which is a particulate gellant and gels a liquid largely due to electrical charges for the attraction between its particles to form a chain network[19], all the other substances gellifying HH are hydrophilic in nature.

The mechanism of gel formation appears to be quite complicated and several factors like solubility parameter, structural characteristics, nature of substituent groups, ease of hydrogen bonding, degree of substitution, gellant molecular weight, pH etc. appear to be operative. It is not possible at this stage to give some concrete reasons and explanations as to why a particular gellant is effective for a fuel. Nevertheless, the present study has identified the above mentioned parameters to be of crucial importance in formulating gelled fuels.

The results on gelation of MMH (Table 1) clearly reveal that besides MCs only two gellants namely HPMC and ASPAA could gellify it whereas quite a good number of gelling agents are successful for HH and UDMH. No single gellant except HEC, is found to gel hydrazine and its derivatives. Both HPMC and ASPAA appear to be very good gellants as they bring about gelation at a very low concentration and short gelation time. It is observed that these gels have a very good consistency and start flowing when subjected to agitation.

A comparison of the results show that MCs gellify both MMH and UDMH but completely fail to develop even the change in viscosity of HH. LS1MC gellifies UDMH within 20 min at a critical concentration of 4.48 wt per cent whereas MMH needs 9 wt per cent of it and yet requires a longer gelation time. Similar results are obtained when LS2MC is used. A nearly two fold increase in concentration and much higher gelling time for MMH in comparison to UDMH systems, have been recorded. It seems quite surprising that both the LSMCs are of same origin but the LS2MC works at a higher concentration. A closer look on the viscosity values of their 2 per cent aqueous solutions reveals slight difference which explains their behaviour. Further, HSMC gels UDMH at a concentration of 2 wt per cent within 10 minutes whereas 8 wt per cent of it is needed to gellify MMH with 18 fold more time required for gelation. This variance in effectiveness levels of MCs may be attributed to the presence of an extra -CH_3 group in UDMH which will impart more basic character to it. Thus, this electron repelling group will facilitate more hydrogen bonding with MC molecules leading to a reduction in critical concentration of the gellant. Currently, MMH/nitrogen tetroxide (NTO) liquid bipropellant system is being widely used as fuel in rocket engines. Studies[4,10] have indicated that aluminized MMH/NTO propellant system may enable planetary missions that would just not be feasible with virgin bipropellant system. Further, a payload increase to the extent of 35 per cent appears to be possible if the space shuttle's solid rocket booster is replaced with a MMH/NTO/*Al* liquid rocket booster[7]. It has also been shown that presence of metal can reduce combustion instabilities in fuels 20 too. Keeping these features in view, the gelation process of MMH has been investigated in some detail with different grades of effective gellants and metal content.It has been found that the gellants like LS2MC and HSMC require unexpectedly longer duration for gelation of MMH at their critical concentrations. In order to understand this peculiar behaviour, efforts have been made to study the effect of marginal variation in gellant concentration from its critical value on gelation time of MMH. The results are summarized in Table 2. In the case of LS1MC, even 1 wt per cent increase brings down the gelation time to half of its value whereas a 2 wt per cent hike in LS2MC level drastically reduces the time from 30 hr to merely 45 min. Similar trend has also been obtained for HSMC where gelation time could be brought down from 3 hr to just 6 min by a 2 wt per cent increase in concentration. This seems to be obvious as the increase in gellant concentration would hasten the gelation process irrespective of its mechanism.

Table 2. Gelation of MMH using methyl cellulose as gellant

Gellant	Concentration (wt %)	Gelation time (min)
LS1MC	0 - 8.0	x
	9.0	27
	10.0	13
LS2MC	0 - 10.0	x
	11.0	1800
	12.0	1680
	13.0	45
HSMC	0 - 7.0	x
	8.0	180
	9.0	20
	10.0	6

Table 3. Effect of HPMC molecular weight on gelation of MMH

Gellant	Concentration (wt %)	Gelation time (min)
HPMC4K	0 - 4.5	x
	5.0	19
	5.5	15
	6.0	12
HPMC15K	0 - 3.0	x
	3.5	15
	4.0	13
	5.0	7
HPMC100 K	0 - 2.50	x
	2.75	12
	3.00	11
	4.00	9

Table 3 presents the data showing the effect of HPMC molecular weight and extent of gellant content on MMH gelation time. It is clear from the results that critical concentration shows a dependence on molecular weight of the gellant, i.e. the higher the molecular weight, the lesser the gellant requirement as well as the gelation time. An increase in gellant concentration is also observed to reduce the time required for gel formation in all these cases too, as discussed above.

As mentioned earlier (Table 1) the synthetic gellant forMMH gelation. However, other gellants of the same series have also been found to be quite effective. The critical concentrations of these gellants and the corresponding gelation times are given in Table 4. It is apparent that these chemicals gellify MMH almost instantaneously within a very low (wt per cent) concentration level.

Table 4. Critical concentrations of synthetic gellants for MMH gelation

Gellant	Critical-concentration (wt%)	Gelation time (min)
ASPAA I	2.10	1.0
ASPAA II	1.75	1.0
ASPAA III	2.65	1.0

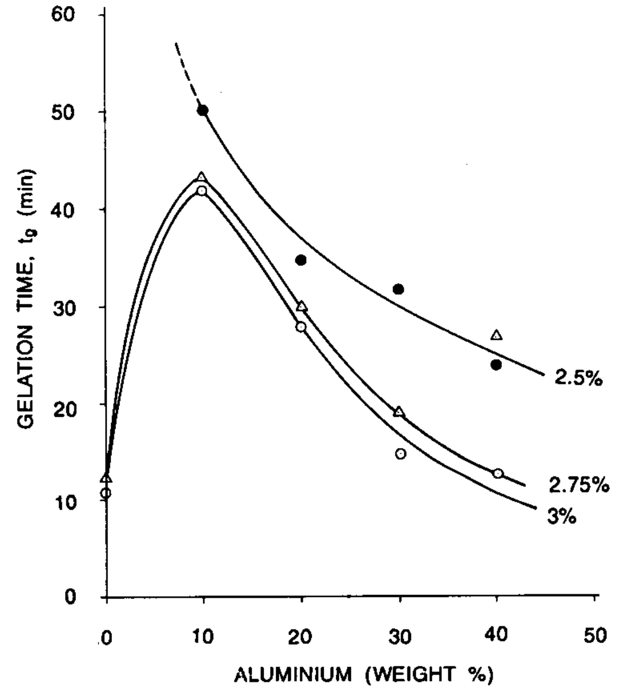

Figure 1. Variation of gelation time with degree of aluminization in MMH-HPMC100K system at different gellant concentrations.

3.2 Metallized MMH Gelled Systems

The effect of aluminium content (0-40 wt per cent) in the MMH-HPMC100K system on gelation time t_g has been depicted in Fig.1 for varying gellant concentrations. It is seen that t_g is inversely related with gellant concentration as the curves shift upward with the reduction in gelling agent content in the gel. It is also clearly observed that t_g increases with level of metallization to give its maximum value corresponding to 10 wt per cent of metal loading in all the cases. Further increase in *Al* content in the gel results in decrease in t_g values at least up to 40 wt per cent of metal loading covered in the present study. Moreover, it is also apparent from Fig. 1 that the critical gellant concentration for virgin MMH-HPMC100K gel is 2.75 wt per cent which may be brought down to 2.5 wt per cent on metallization.

The reduction in t_g with gellant concentration is understandable as the extent of network formation and hydrogen bonding increases which quickens the physical gelation process. The increase in the t_g value on metallization may be attributed to the fact that the coarse metal particles initially hinder the gel network formation merely due to dilution effect and then adversely affect the viscosity build-up by virtue of their shearing action on the system under the influence of gravity. However, it is also important to note that the ultrafine metal powders of colloidal dimensions are known to assist in gelation[21] due to Vander Waal's forces. All metal powders have such particle size component in some measure. At higher metal loadings, the role of ultrafine metal particles may influence the net gelation time to a considerable extent. Further, with increasing extent of metallization more and more liquid fuel is going to be used up in wetting the metal particles and thus the 'effective' gellant concentration for gelling the base propellant increases. This would obviously lead to reduction in t_g at higher metal content level (> 10 wt per cent), as observed. It may be mentioned here that higher metal loadings will also reduce the shearing action of metal particles due to increase in their population density, facilitating the gel build up.

Figure 1 further reveals that at an aluminization level of 30 wt per cent and beyond in MMH-HPMC100K system, the t_g approaches close to that of its virgin gel which may further come down with additional metallization. Similar results have also been reported in literature for other heterogeneous systems[5]. This may lead to the conclusion that the role played by the ultrafine metal particles as a particulate gellant in conjunction with the chemical one used, is quite important and decisive in the gelation process of heterogeneous fuel systems.

Table 5. Effect of aluminium content on gelation time of MMH fuel using different grades of HPMC gellant.

Aluminium (wt%)	MMH gelation time (min) with gellant HPMC								
	100 K (wt %)			15K (wt %)			4K (wt %)		
	2.5	2.75*	3.0	3.0	3.5*	4.0	4.0	4.5	5.0
0	x	12	11	x	15	13	x	x	19
10	50	43	42	21	20	15	17	9	9
20	35	30	28	28	22	11	14	11	9
30	32	19	15	x	27	20	17	14	10
40	24	27	13	x	33	16	20	17	9

x No gelation occurs

* Critical concentration for virgin gel.

It is pertinent to note that the molecular weight of HPMC gellant influences its critical concentration level and gelation time. Further, the degree of metallization has also been found to affect the gelation process at varying gellant concentrations. The experimental results are recorded in Table 5. It is seen that the critical concentration of gellants HPMC100K, 15K and 4K for aluminized gels could be brought down by 9,14 and 20 wt per cent respectively. This reflects that highly metal-loaded gel would require reduced quantities of gellant. However, from stability and long term storage point of view, the use of a slightly higher than critical concentration may be desirable.

The results recorded on the dependence of t_g on magnesium level in MMH-HPMC100K system at varying gellant concentrations have been presented in Fig. 2. The general trend in this case is also similar to that exhibited by aluminized gels. However, the effect on gelation process produced by ultrafine particles in the case of *Al* would be neglible here as the *Mg* particle size used is coarse (60 µm). This factor would also cut down the shearing effect of magnesium powder. This explains the reduced t_g values obtained in case of magnesium containing gels. An important observation for magnesium containing gels is that the critical gellant concentration gets reduced by nearly 18 per cent which is just double of that for aluminized gels. It is also noteworthy that the t_g shown by metallized gels containing 20 wt per cent and beyond of *Mg* drops to a level even below to that of virgin gel.

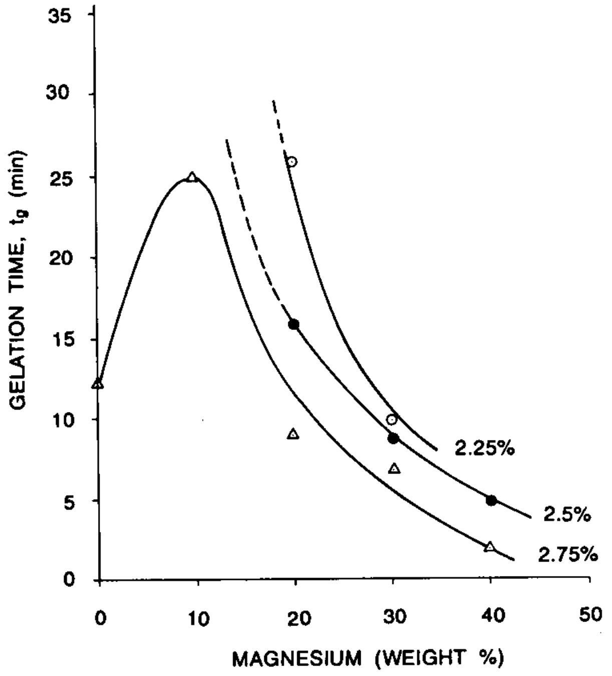

Figure 2. Dependence of gelation time on magnesium level in MMH- HPMC100K system at varying gellant concentrations.

The influence of the magnesium content on gelation time of MMH at fixed gellant concentrations has also been investigated using low and high substituted methyl cellulose, with a view to confirming the general validity of findings in the case of HPMC gelled systems. The results have been plotted in Fig. 3. As reported earlier, the HSMC has a critical concentration of 8 wt per cent to effect MMH gelation which can be reduced to 7 wt per cent corresponding to 30 wt per cent metal content. Below this *Mg* level in the MMH-HSMC system, the gelation does not occur at all. This may lead to the conclusion that the higher population density of the metal particles in the bulk aids in the gelation process possibly because of the substantial presence of fine component of metal particles and increased effective gellant concentration in fuel, as a sizable quantity of

MMH will now be used in wetting metal particles. It is seen that the gelation time gets drastically reduced on metallization, in this case also.

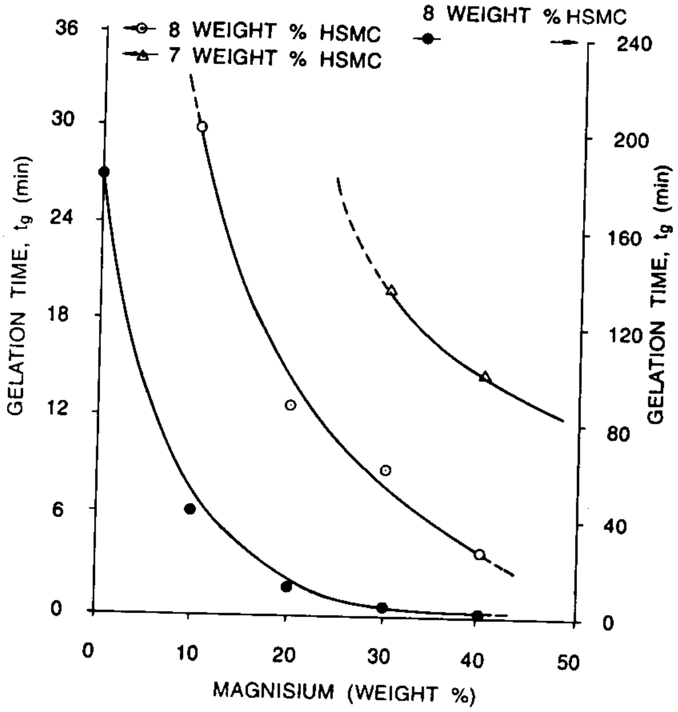

Figure 3. Effect of magnesium content on gelation time of MMH-MC system.

3.3 Ageing Behaviour of MMH Gelled Systems

The MMH gels prepared with gellants like HPMC100K and ASPAA- II were stored for a period of three months in QF stoppered specimen tubes and observations regarding physical changes were made. During this period of storage studies, the ambient temperature varied between 17 and 35 °C. No physical changes in the virgin MMH gels were observed suggesting that these gels are quite stable and firm in nature for sufficiently long time. The level of the liquid premarked on the specimen tube soon after the mixing of the ingredients remained unchanged, indicating that the gelation has prevented vaporization losses of the fuel almost completely.

The aluminized gels of MMH-HPMC100K system with different gellant concentrations and metal content were also stored and observed. The experimental results showed (data not reported) that a homogeneous dispersion of the metal could easily be achieved initially. Subsequently, inhomogeneity in the distribution of metal particles in the bulk was observed. It may be reported that the metallized gels prepared using the critical concentration of gellant for the parent fuel showed lesser settling of metal powder in comparison with other gellant levels. It is also worth mentioning that with increase in metal content in the gel, the settling of metal powder is curtailed to a great extent, e.g. 30 wt per cent *Al* containing gels have shown a metal settling of the order of only < 3 wt per cent. However, all the metallized gelled fuels could conveniently be reconstituted to a homogeneous system simply by remixing them. Yet another but practical approach is to prepare the gels with tailored yield stress to prevent the metal settling completely.

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